

CLAIMS

1. A microporous crystalline material of zeolitic nature, characterized in that the material has, in a anhydrous state calcinated at temperatures between 300°C and 800°C, an X-ray diffraction pattern according to

d(Å)	(I/I ₀) * 100
32.82 ± 0.02	vs
11.97 ± 0.03	w
10.05 ± 0.04	w
9.39 ± 0.06	m
7.05 ± 0.05	w
6.93 ± 0.02	w
6.56 ± 0.05	w
5.64 ± 0.07	w
4.77 ± 0.08	w
4.27 ± 0.04	w
3.98 ± 0.08	w
3.89 ± 0.08	w
3.72 ± 0.03	w
3.53 ± 0.05	w
3.46 ± 0.07	w
3.34 ± 0.06	w
2.90 ± 0.08	w

wherein

w is a weak relative intensity between 0 and 20%;

m is an average relative intensity between 20 & 40%;

s is an average relative intensity between 40 and 60%;

vs is an average relative intensity between 60 and 100%

and in that it has an empirical formula



wherein

x has a value less than 0.2;

y has a value less than 0.1:

M is at least one +n charge inorganic cation,

X is at least one chemical element with a +3 oxidation state, preferably selected from the group consisting of Al, Ga, B, Cr and Fe;

Y is at least one chemical element with a +4 oxidation state, preferably selected from the group consisting of Ge, Ti, Sn and V.

2. A crystalline material according to claim 1, characterized in that

x has a value less than 0.1, preferably less than 0.02,

y has a value less than 0.05, preferably less than 0.02.

3. A crystalline material according to claim 2, characterized in that x has the value of 0.

4. A crystalline material according to claim 1, characterized in that M is H.

5. A crystalline material according to claim 1, characterized in that it has specific surface characteristics measured by N₂ adsorption-desorption, with an external surface of at least 100 m²g⁻¹, and preferably with more than 400 m²g⁻¹.

6. A process to synthesize the crystalline material of claim 1, characterized in that it comprises

a first step wherein a precursor is prepared by subjecting to heating, with or without stirring, at a temperature between 100 and 225°C, preferably between 125 and 200°C, a reaction mixture that contains

a SiO₂ source,

optionally a GeO₂ source,

optionally a source of at least another tetravalent element Y preferably selected from the group consisting of Ge, Ti, V and Sn,

optionally a source of at least another trivalent element X preferably selected from the group consisting of Al, B, Ga, Fe and Cr,

an organic cation 1-methyl-1,4-diazabicyclo[2.2.2]octane,

and water,

wherein the reaction mixture has a composition, in terms of molar ratios of oxides, comprised in the ranges of

$\text{ROH}/\text{SiO}_2=0.01-1.0$, preferably 0.1-1.0,

$\text{M}_{1/n}\text{OH}/\text{SiO}_2=0-1.0$, preferably 0-0.2,

$\text{X}_2\text{O}_3/\text{SiO}_2=0-0.1$, preferably 0-0.05, and more preferably 0-0.01,

$\text{YO}_2/(\text{YO}_2+\text{SiO}_2)$ less than 1, preferably less than 0.1,

$\text{H}_2\text{O}/\text{SiO}_2=0-100$, preferably 1-50,

wherein

M is at least a +n charge inorganic cation;

X is at least a trivalent element preferably selected from the group consisting of Al, B, Ga, Fe and Cr;

Y is at least a trivalent element preferably selected from the group consisting of Ge, Ti, Sn and V;

R is an organic cation, preferably 1-methyl-1,4-diazabicyclo[2.2.2]octane

until crystallization of the reaction mixture is achieved;

a second stage wherein the precursor is subjected to swelling in a swelling solution in order to obtain a swollen laminar material;

a third stage wherein the swollen laminar material is washed and dried in order to obtain a dry swollen solid;

a fourth stage wherein the dry swollen solid is subjected to at least partial delamination in water in order to obtain a suspension that contains a material at least partially delaminated;

a fifth stage wherein the material at least partially delaminated is separated from the suspension;

a sixth stage wherein the organic wastes are removed from the at least partially delaminated material, by means of a treatment selected from the group consisting of cationic exchange, calcination and combinations thereof.

7. A process according to claim 6, characterized in that the organic cation 1-methyl-1,4-diazabicyclo[2.2.2]octane is added in the form of a hydroxide and another salt, preferably halide, to the reaction mixture.

8. A process according to claim 6, characterized in that an amount of crystalline material, preferably with the characteristics of the material of claim 1 as the crystallization promoter is added to the reaction mixture, said amount being comprised between 0.01 to 15%, preferably 0.05 to 5% by weight referred to the total silica added.

9. A process according to 6, characterized in that the precursor has an X-ray diffractogram that comprises values corresponding to

d(Å)	(I/IO)*100	d(Å)	(I/IO)*100
11.22±0.02	vs	3.60±0.08	s
10.10±0.03	w	3.52±0.05	vs
8.81±0.05	w	3.42±0.06	s
7.05±0.01	w	3.36±0.04	s
6.30±0.01	m	3.32±0.05	w
5.60±0.02	w	3.30±0.01	w
5.28±0.05	s	3.14±0.07	w
4.98±0.06	s	3.10±0.02	w
4.72±0.01	w	3.09±0.03	w
4.38±0.02	s	3.01±0.01	w
4.21±0.02	s	2.81±0.04	w
3.90±0.03	w	2.61±0.04	w
3.83±0.08	m	3.51±0.05	w

3.73±0.07	m	2.48±0.09	w
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wherein

w is a weak relative intensity between 0 and 20%;

m is an average relative intensity between 20 & 40%;

s is an average relative intensity between 40 and 60%;

vs is an average relative intensity between 60 and 100%.

10. A process according to claim 6, wherein the swelling solution with which the dry swollen solid is obtained, contains organic molecules that are intercalated in order to produce a separation of sheets of the precursor.

11. A process according to claim 10, characterized in that the organic molecules used in the swelling solution with which the dry swollen solid is obtained are selected between molecules that have a proton acceptor group and a hydrocarbonaceous chain.

12. A process according to claim 10, characterized in that the organic molecules used in the swelling solution with which the dry swollen solid is obtained are selected from among alkylammonium molecules, with a number of carbon atoms between 4 and 24, preferably between 16 and 19.

13. A process according to claim 10, characterized in that the organic molecules used in the swelling solution with which the dry swollen solid is obtained are molecules of cetyltrimethylammonium chloride (CTMA⁺).

14. A process according to claim 6, characterized in that the swelling solution comprises
 a suspension of the precursor in a solid suspension between 10 and up to 50% by weight;
 a cetyltrimethylammonium hydroxide solution (OH^- , Br^-) between 10 up to 50% by weight;
 a tetrapropylammonium solution (TPA^+) (OH^- , Br^-) between 20 up to 60% by weight.

15. A process according to claim 6, characterized in that the swelling solution comprises

a suspension of the solid precursor up to 20% by weight;

a cetyltrimethylammonium hydroxide solution (OH^- , Br^-) up to 29% by weight;

a tetrapropylammonium solution (OH^- , Br^-) up to 40% by weight;

with a weight ratio of precursor suspension :
 cetyltrimethylammonium hydroxide (OH^- , Br^-) solution :
 tetrapropylammonium (OH^- , Br^-) solution of 27:105:33.

16. A process according to claim 13, characterized in that the dry swollen solid has X-ray diffraction values corresponding to.

$d(\text{\AA})$	$(I/I_0) * 100$	$d(\text{\AA})$	$(I/I_0) * 100$
37.89 ± 0.02	vs	4.71 ± 0.03	w
14.50 ± 0.02	s	4.23 ± 0.04	m
12.50 ± 0.03	m	4.14 ± 0.08	m
11.73 ± 0.01	w	3.95 ± 0.09	m
10.01 ± 0.04	w	3.86 ± 0.08	m
7.66 ± 0.05	w	3.82 ± 0.09	m
7.36 ± 0.03	w	3.51 ± 0.08	m
6.99 ± 0.06	w	3.43 ± 0.08	w
6.55 ± 0.05	w	3.31 ± 0.05	w
5.98 ± 0.01	w	3.18 ± 0.07	w
5.59 ± 0.08	w	2.88 ± 0.09	w
5.11 ± 0.04	w		

wherein

w is a weak relative intensity between 0 and 20%;

m is an average relative intensity between 20 & 40%;

s is an average relative intensity between 40 and 60%;

vs is an average relative intensity between 60 and 100%.

17. A process according to claim 6, characterized in that the fourth step is carried out at least by means of a technique selected from the group consisting of mechanical stirring, ultrasound, spray-drying, lyophilization and combinations thereof.

18. A process according to claim 6, characterized in that the suspension that contains the at least partially delaminated material is subjected to flocculation.

19. A process according to claim 6, characterized in that, in the sixth stage, the at least partially delaminated material is calcinated in an air flow, at a temperature between 300°C and 800°C for at least 3 hours.

20. A process according to claim 19, characterized in that the temperature is between 400°C and 600°C.